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Effect of Convection on Primary Dendrites: Observations from Ground-based and Space Station Processed Samples.

Masoud Ghods – Cleveland State University

Mark Lauer – The University of Arizona

Surendra N. Tewari – Cleveland State University

Richard N. Grugel – Marshall Space Flight Center

Robert E. Erdman – The University of Arizona

David R. Poirier – The University of Arizona

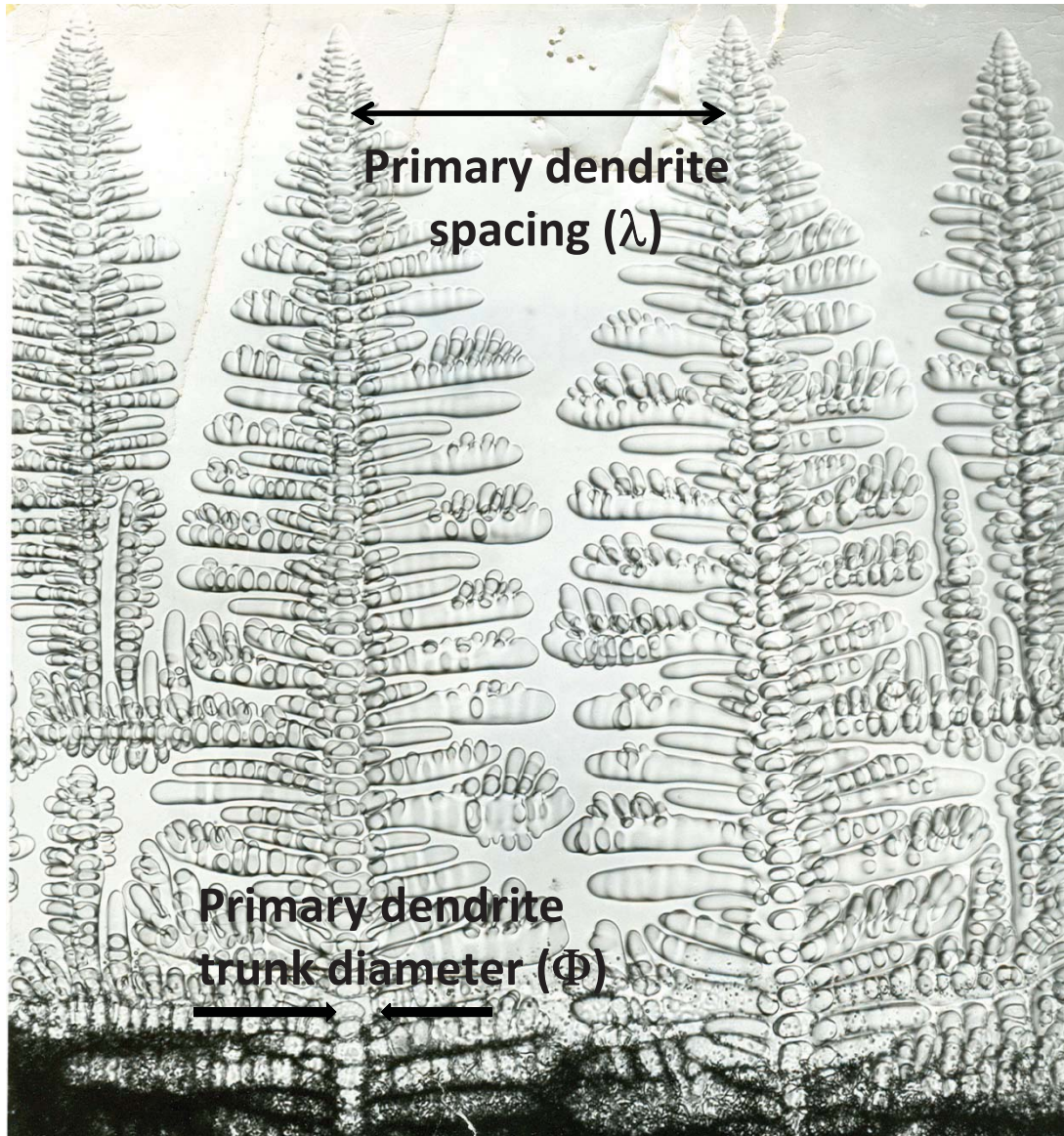
Collaborative Effort between NASA and European Space Agency : Program MICAST

*(Microstructure Formation in Castings of Technical Alloys under Diffusive and
Magnetically Controlled Convective Conditions)*

Outline

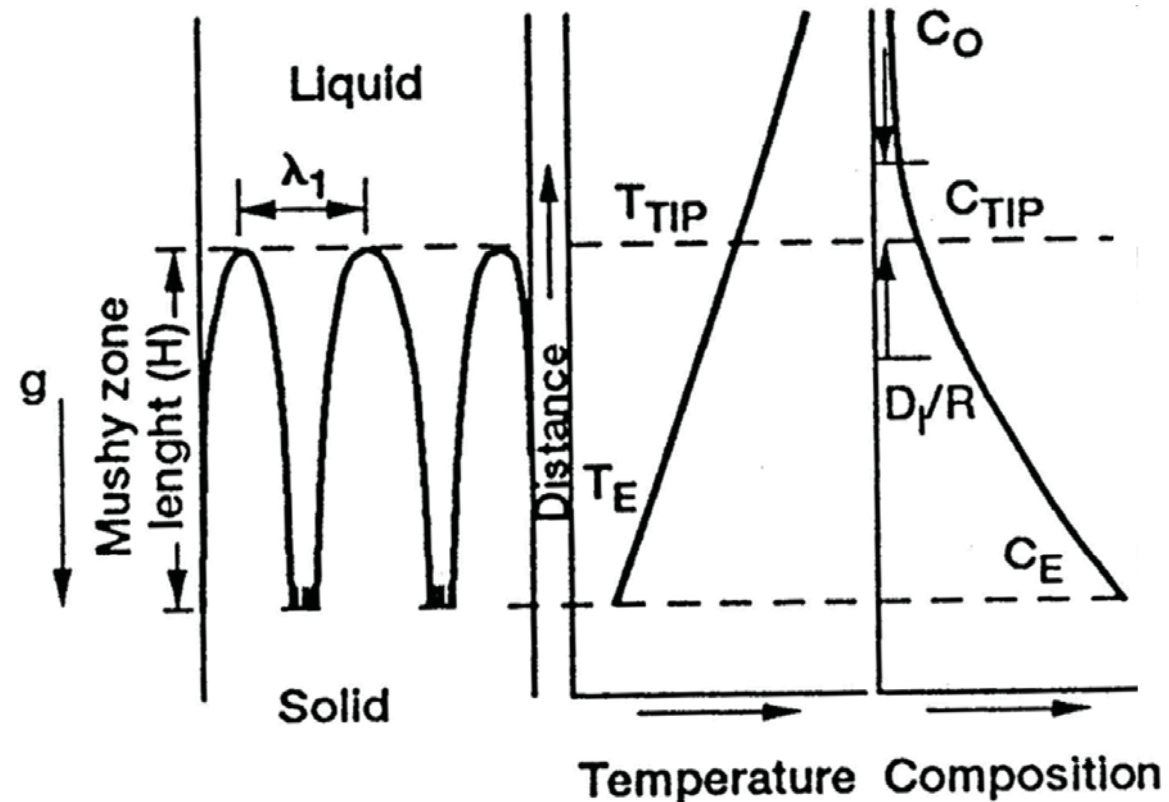
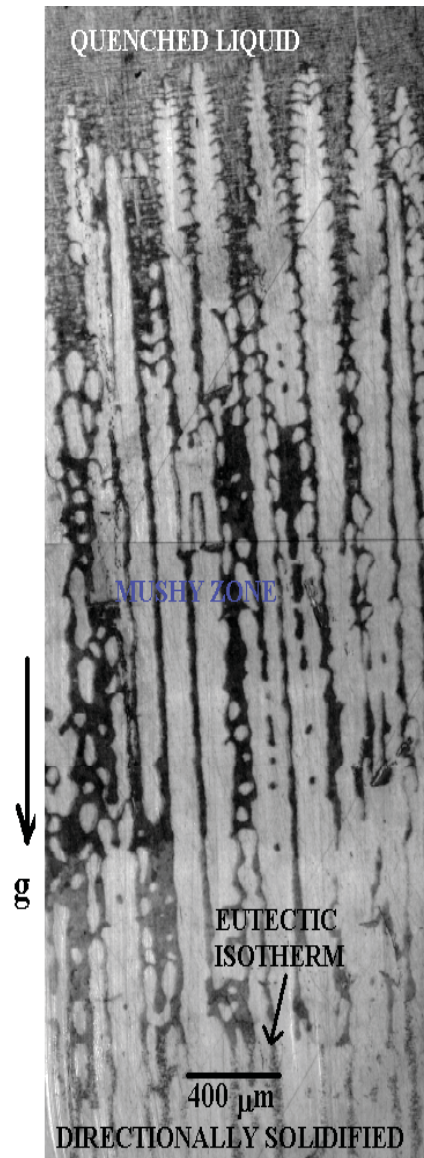
- Introduction: Directional solidification(DS)
 - Dendritic array (why “low gravity”?)
 - Previous low-g dendritic array DS
- Purpose
- Ground-based and low gravity results
 - Primary dendrite spacing
 - Primary dendrite trunk diameter
 - Radial macro-segregation
- Surprises

Dendritic array morphology depends upon DS processing parameters: G_I , R , C_o , *Convection*??



1. Primary dendrite arm spacing (λ): Extensive literature (SCN/Metals)
2. Secondary/tertiary arm spacing: Extensive-SCN/Metals
3. Dendrite tip radius: SCN/limited (Al-Cu, Pb-Au, Pb-Pd)
4. Primary dendrite trunk diameter (Φ): Limited (Esaka:Thesis-86, Grugel: 92/95)

Primary dendrite array during directional solidification in a positive thermal gradient



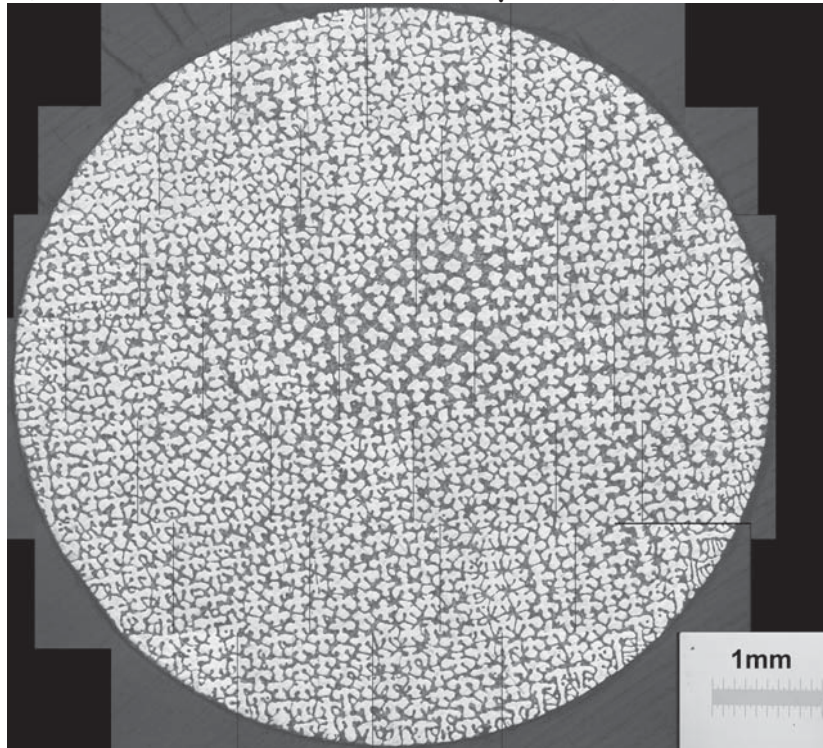
- Thermal profile: stabilizing
- Solutal profile:
 - destabilizing (solute enrichment reduces density, $\beta_c > 0$): Pb-6Sb)
 - stabilizing (solute enrichment increases density, $\beta_c < 0$, Al-19Cu)

Natural convection is inevitable during dendritic DS on earth

-Thermally stable

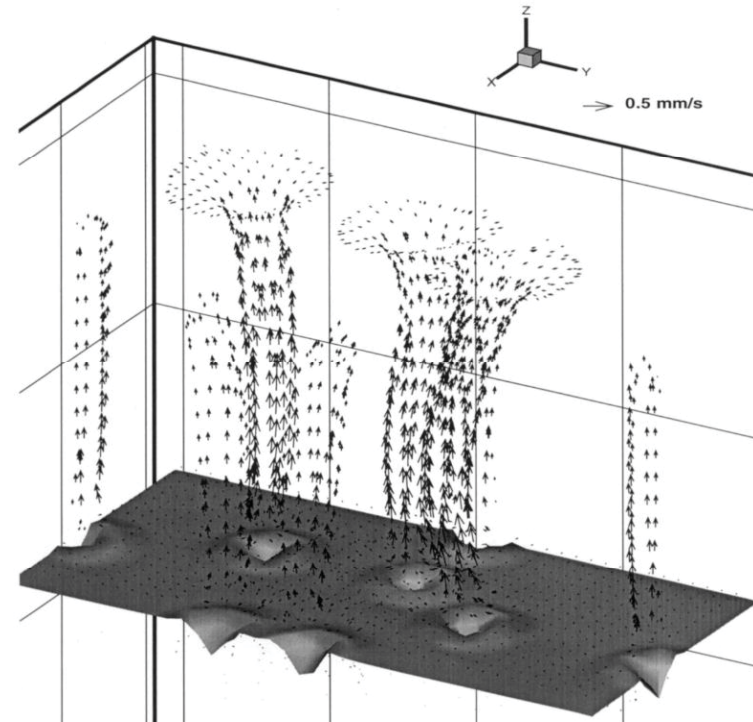
-Solutally unstable

(Pb-5.8Sb: 40 K cm^{-1} , $10 \mu\text{ms}^{-1}$)



Axial macrosegregation

Radial macrosegregation (Freckle)



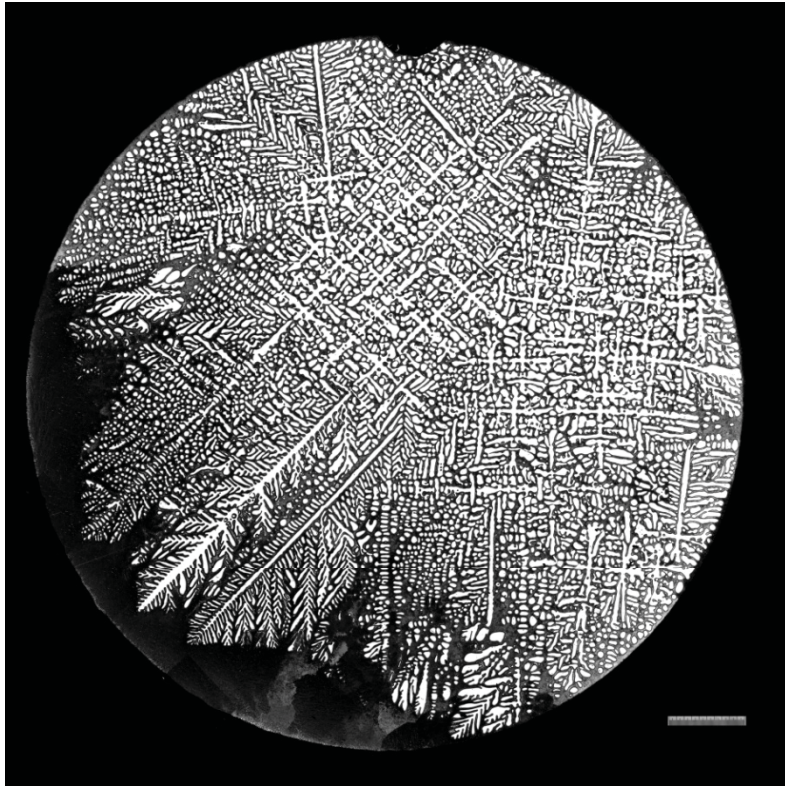
Felicelli, Poirier, Heinrich (1998)

Several numerical models

(Poirier et al., Beckermann et al.)

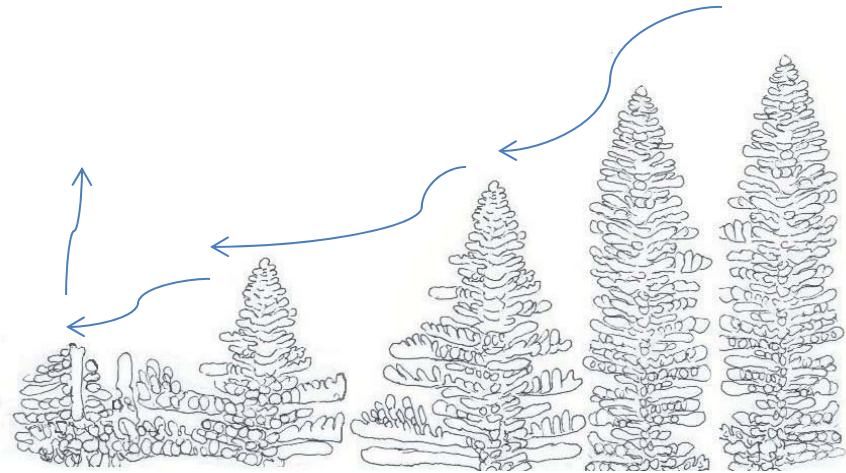
Dendritic array growth under diffusive solutal and thermal transport only possible in the absence of “g”

Natural convection is inevitable during dendritic DS on earth



Radial macrosegregation
Dendrite steepening

- Thermally stable
 - Solutally stable
- (Al-19 Cu: 81 K cm^{-1} , $10 \mu\text{ms}^{-1}$)



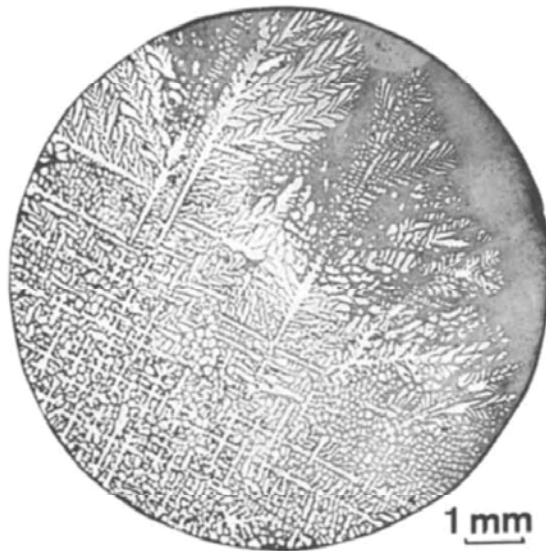
No analytical or numerical models

Dendritic array growth under diffusive solutal and thermal transport only possible in the absence of "g"

Natural convection decreases primary dendrite spacing

(M.D. Dupouy, D. Camel and J.J. Favier, Acta. Metall. Mater. Vol. 37, No. 4, pp. 1143-1157, 1989)

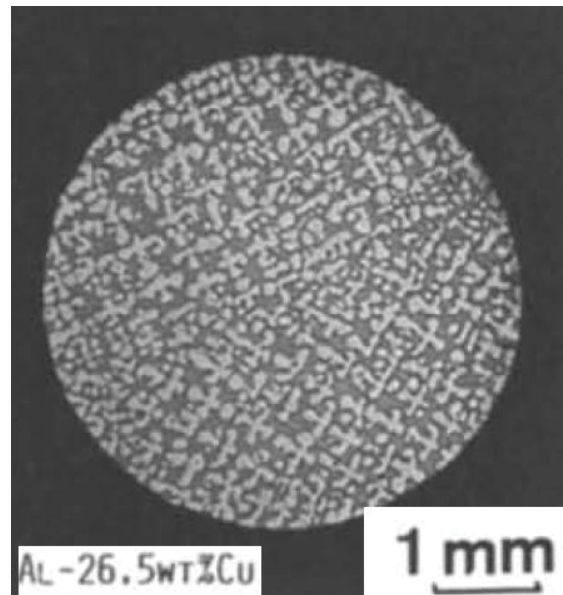
Al-26.5 wt% Cu, 30 K cm⁻¹,
4.2 μm s⁻¹



Terrestrial: Solutally stable,
thermally stable mode

Primary spacing → 450 ± 20 μm

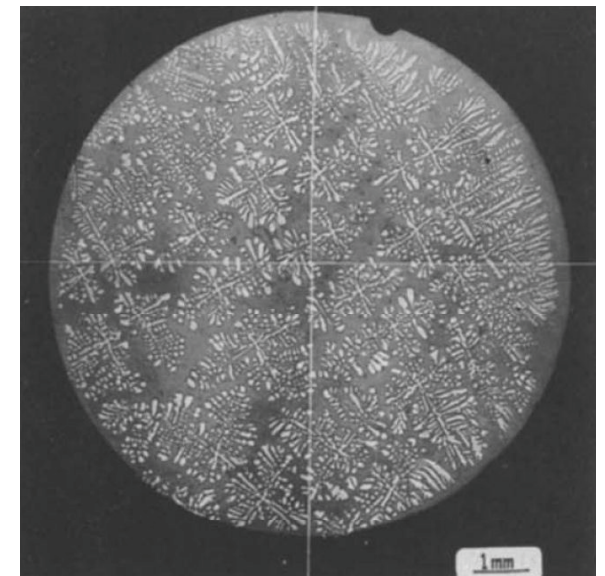
Al-26.5 wt% Cu, 25 K cm⁻¹,
4.2 μm s⁻¹



Terrestrial: Solutally unstable,
thermally stable mode

340 ± 10 μm

Al-26.5 wt % Cu, 30 K
cm⁻¹, 4.2 μm s⁻¹



Microgravity:

1540 ± 10 μm

Purpose

MICAST: A systematic analysis of the effect of convection on the microstructural evolution in cast binary, ternary and commercial Al-Si based alloys.

MICAST6



MICAST 7

Re-melt and DS terrestrially grown dendritic mono-crystals of Al-7 wt% Si (9-mm dia, 25 cm long) in μ g.

Advantages:	Minimize Thermo-Solutal Convection
Intent:	Produce Segregation Free Samples Grown Under Diffusion-Controlled Conditions
Purpose:	Better Understand the Relationship between Processing and Microstructure-Development

1. Primary dendrite spacing
2. Primary dendrite trunk diameter
3. Radial macrosegregation

Microgravity processing : Partially remelt and then DS from terrestrially grown dendritic mono-crystal in μg .



(Al-7%Si Single Crystal Dendritic)



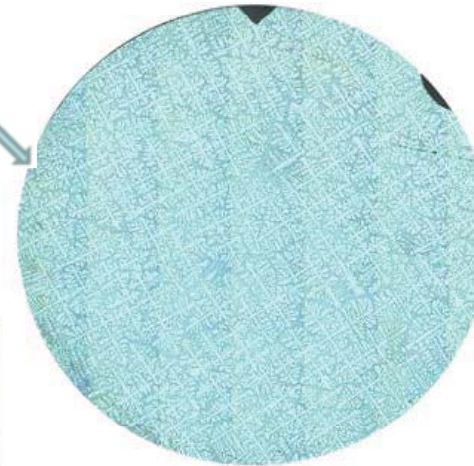
ESA- Sample Cartridge Assembly



ESA_MSL Low Gradient Furnace



NASA_MSSR-1 Flight Rack



Transverse View

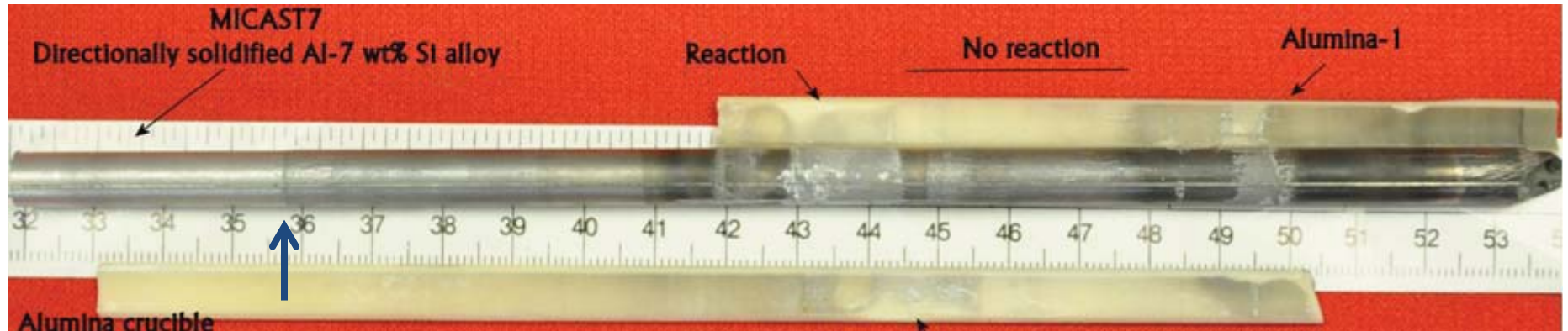
ESA:
Material
Science
Laboratory

Microgravity processed sample MICAST 7



Eutectic Melt Back
/ Isotherm

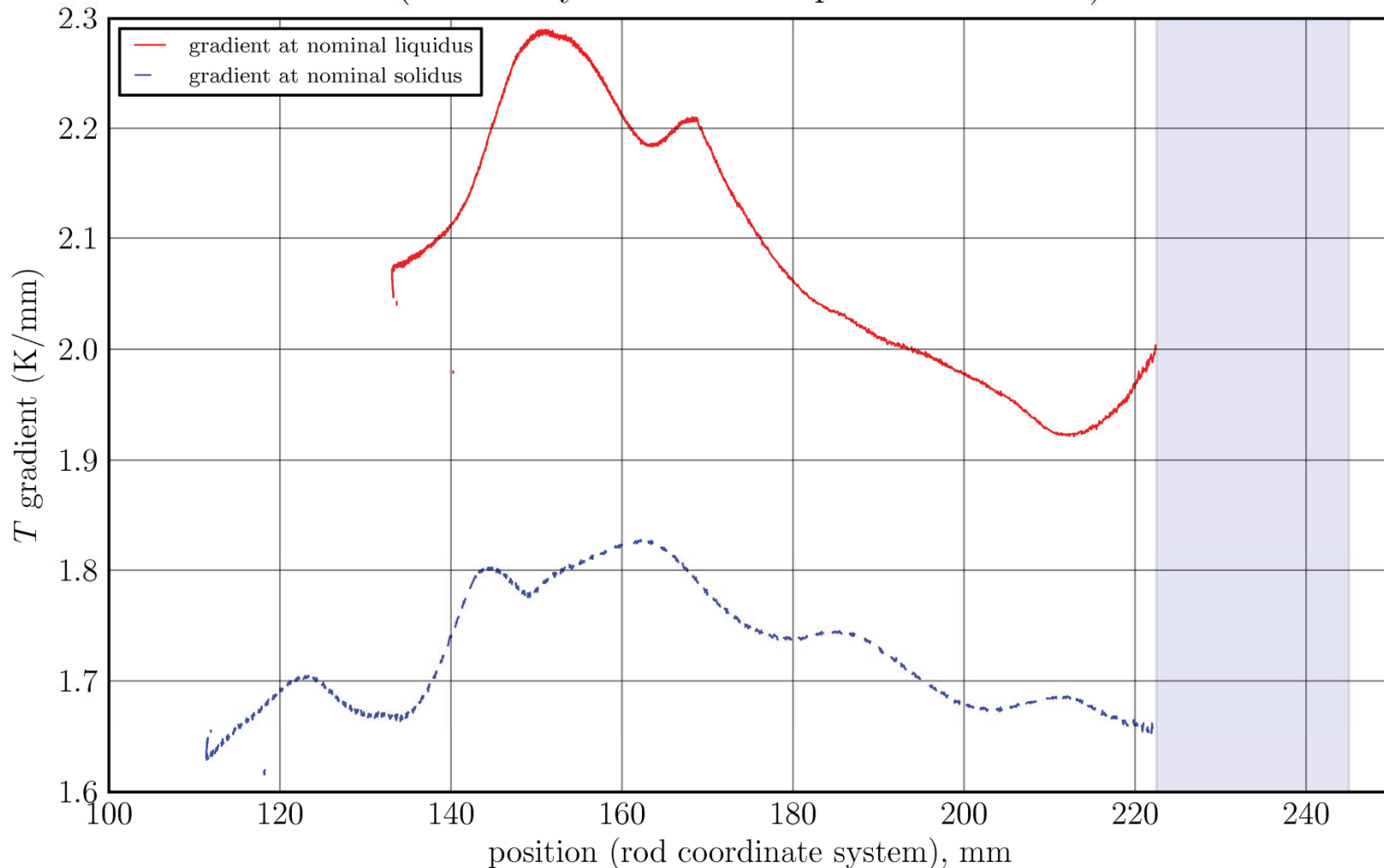
X-ray radiograph of MICAST7



Eutectic Melt Back

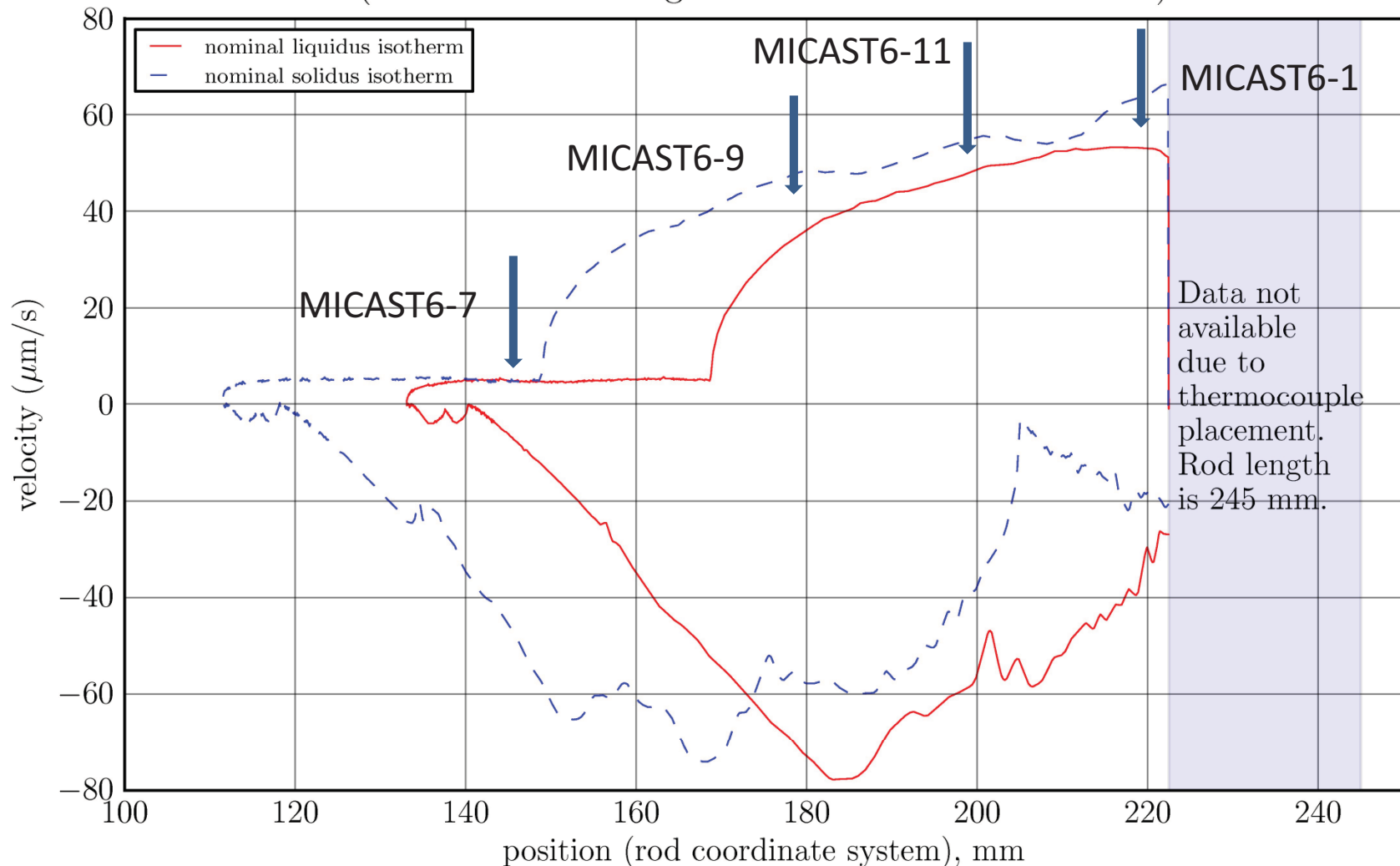
MICAST6: ESA-Low Gradient Furnace (1-hr heat-up, 5-hr hold, $G_l \sim 20 \text{ K cm}^{-1}$): 3.8 cm at $5 \mu\text{m s}^{-1}$, 11.3 cm at $50 \mu\text{m s}^{-1}$

temperature gradients at nominal liquidus and solidus isotherms
(note: only solidification portion is shown)



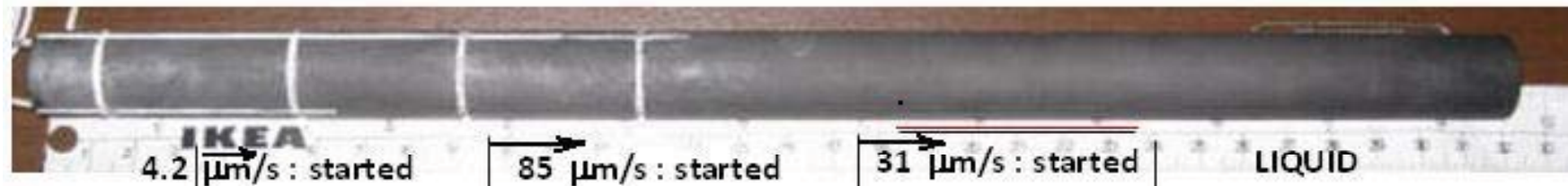
MICAST6: ESA-Low Gradient Furnace (1-hr heat-up, 5-hr hold, $G_1 \sim 20 \text{ K cm}^{-1}$): 3.8 cm at $5 \mu\text{m s}^{-1}$, 11.3 cm at $50 \mu\text{m s}^{-1}$

isotherm velocity vs. position along the Al-Si rod
(note: both melting and solidification are shown)

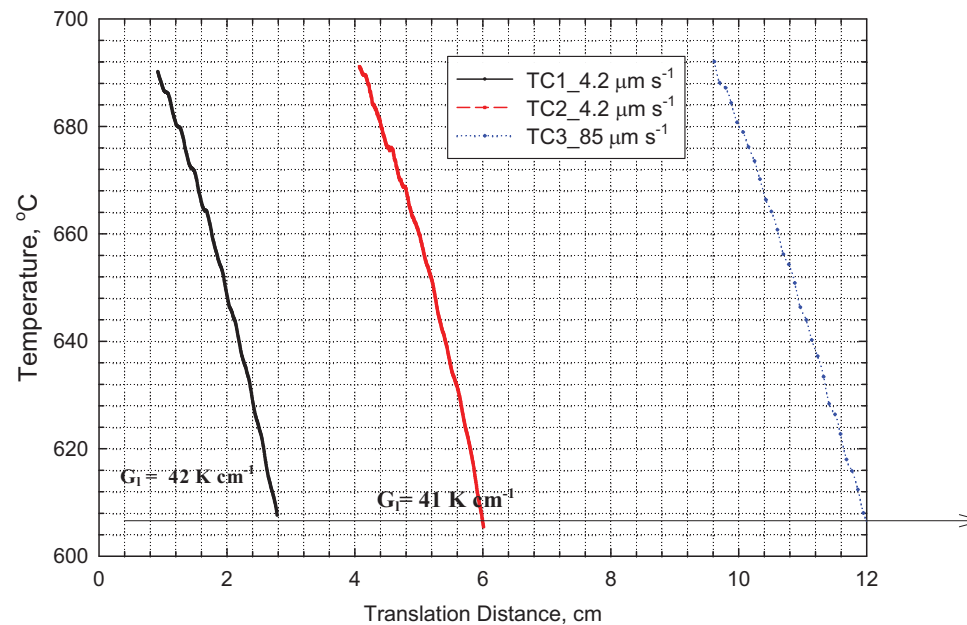


Terrestrial processing

Graphite crucible (~9 mm ID, ~19 mm OD), 10^{-4} torr vacuum



Thermal Gradient at the liquidus temperature
(Al-7%Si, Graphite Crucible, 3 TCs located along crucible length)

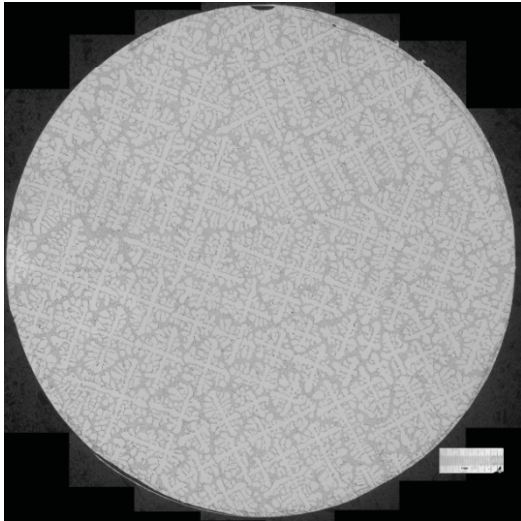


Don't have terrestrial samples which are processed in LGF or LGFQ equivalent hardware under R and G_1 conditions which are identical to MICAST6, MICAST7

Comparison of microstructures: Al-7% Si directionally solidified on ground and on ISS (MICAST6)

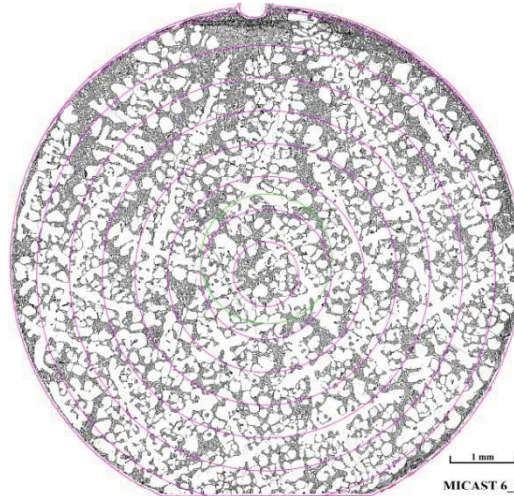
MICAST6 SEED

41 K cm^{-1} , $22 \mu\text{m s}^{-1}$



MICAST6: 20 K cm^{-1}

$5 \mu\text{m s}^{-1}$



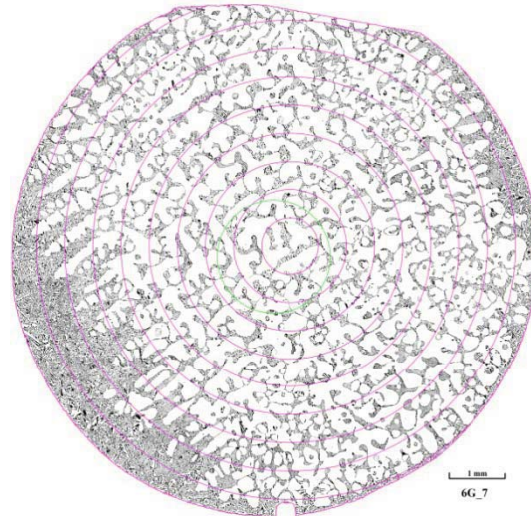
$50 \mu\text{m s}^{-1}$



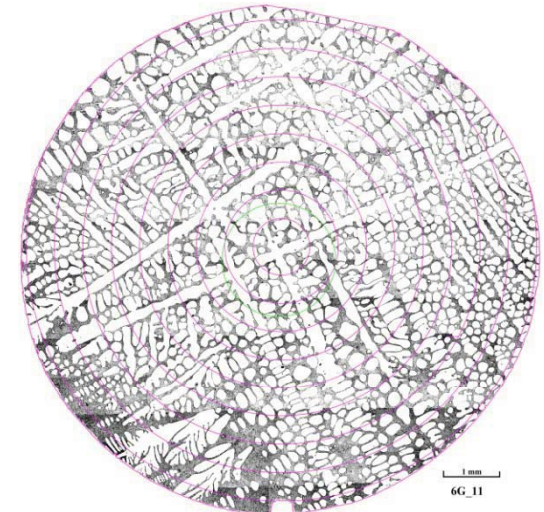
Terrestrial DS:

15 K cm^{-1} →

Convection causes dendrite clustering (steeping) and radial macrosegregation at low thermal gradient and growth speeds during terrestrial DS.



$5 \mu\text{m s}^{-1}$

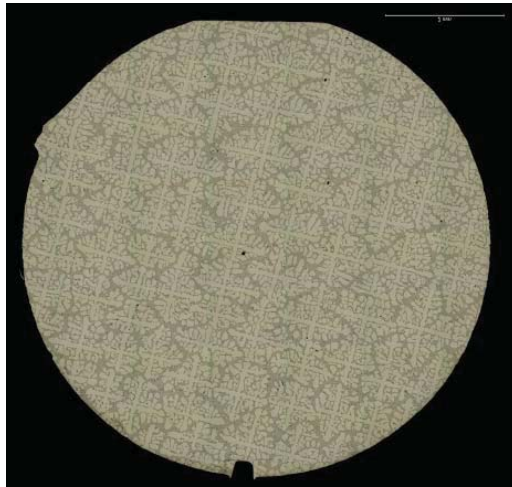


$50 \mu\text{m s}^{-1}$

Comparison of microstructures: Al-7% Si directionally solidified on ground and on ISS (MICAST7)

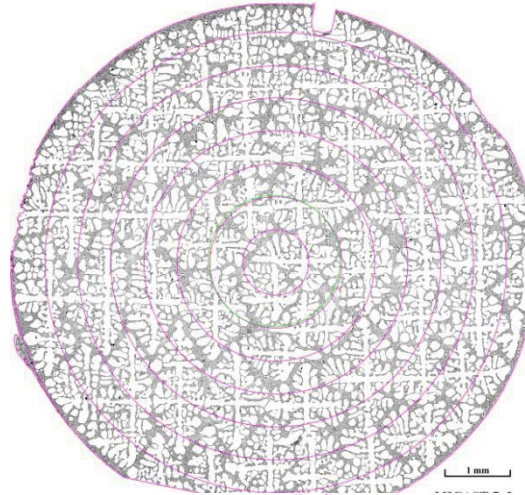
MICAST7 SEED

41 K cm⁻¹, 22 μm s⁻¹



MICAST7: 26 K cm⁻¹

21 μm s⁻¹

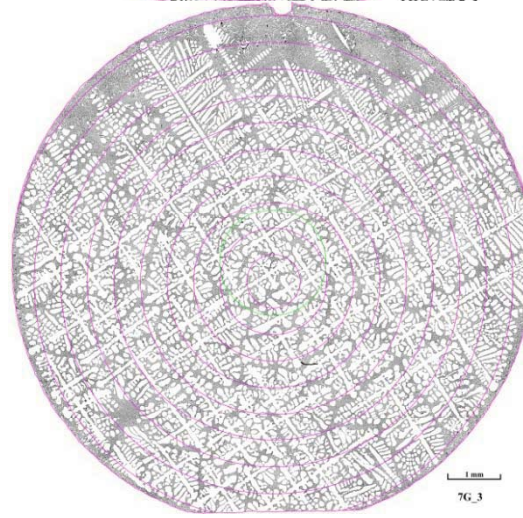


11 μm s⁻¹

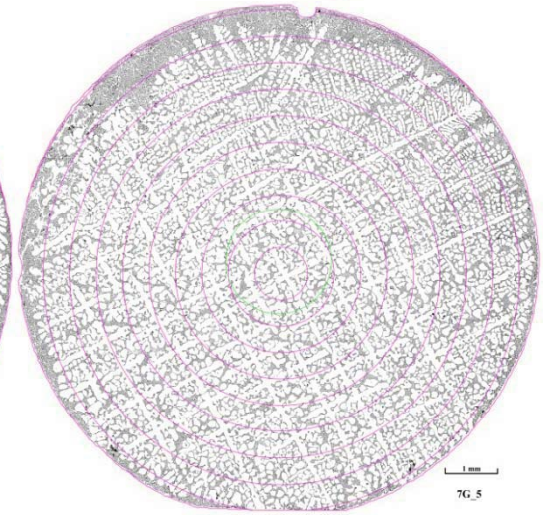


Terrestrial DS:

24 K cm⁻¹ →



23 μm s⁻¹



10 μm s⁻¹

Theoretical models : Primary dendrite arm spacing (Diffusion controlled growth)

- Trivedi (1984)
- Hunt-Lu (1996)

J.D. Hunt and S.Z. Lu, MMT, 1996

$$\lambda' = \frac{\lambda \Delta T_o}{\Gamma k} \quad V' = \frac{V \Gamma k}{D \Delta T_o} \quad G' = \frac{G \Gamma k}{\Delta T_o^2}$$

Measured: λ , G , V

Alloy Properties:

ΔT_o = sol'dn. temp. range, K

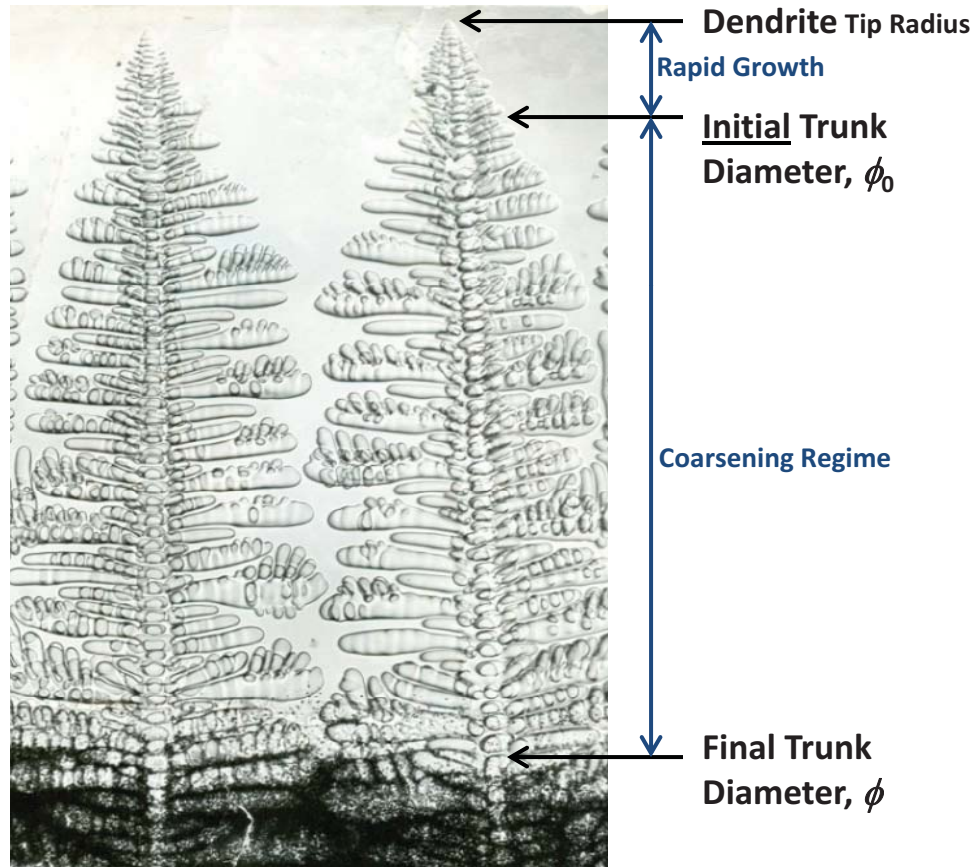
Γ = Gibbs-Thomson coefficient, m K

k = equilibrium partition ratio

D = diffusion coefficient of Si in liquid, m²/s

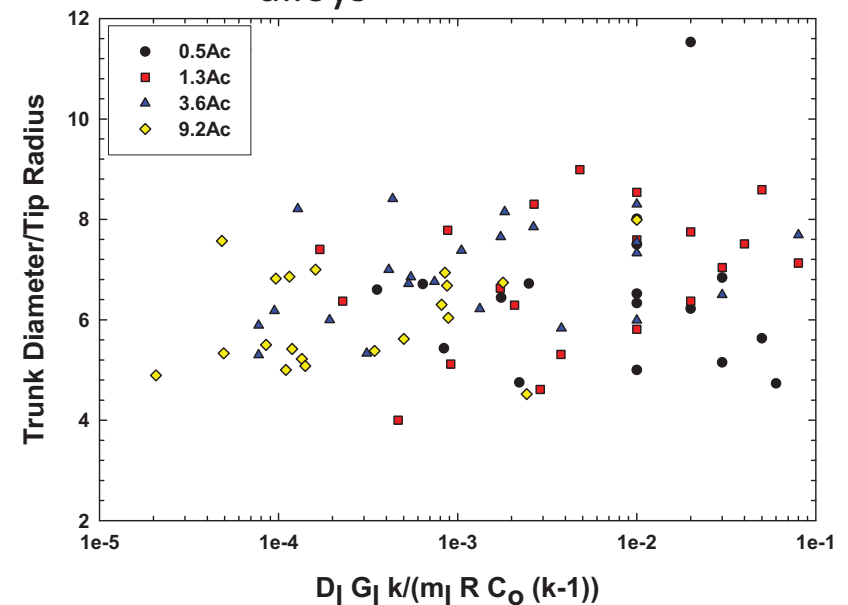
→Theoretical models predict *nearest neighbor spacing*

Theoretical Model: Primary Dendrite Trunk Diameter (ϕ)



“Initial” Trunk Diameter (ϕ_0) Determination

Esaka (1986 Ph.D. Thesis)
Succinonitrile-Acetone
“alloys”



$$\phi_0 = 6.59 \pm 1.3 r_{\text{tip}}$$

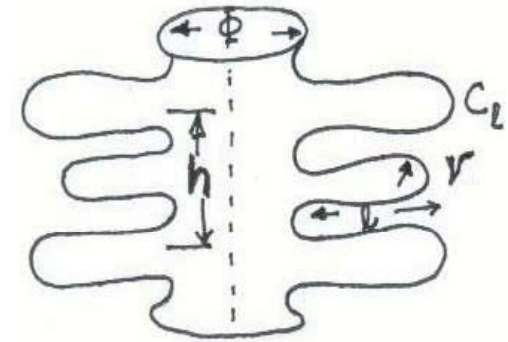
Primary dendrite trunk diameter (ϕ) model

After ϕ_0 the trunk diameter increases via dissolution of secondary arms and redeposition on the trunk until the eutectic.

Kirkwood ripening-model (1985) is used.

Assumptions:

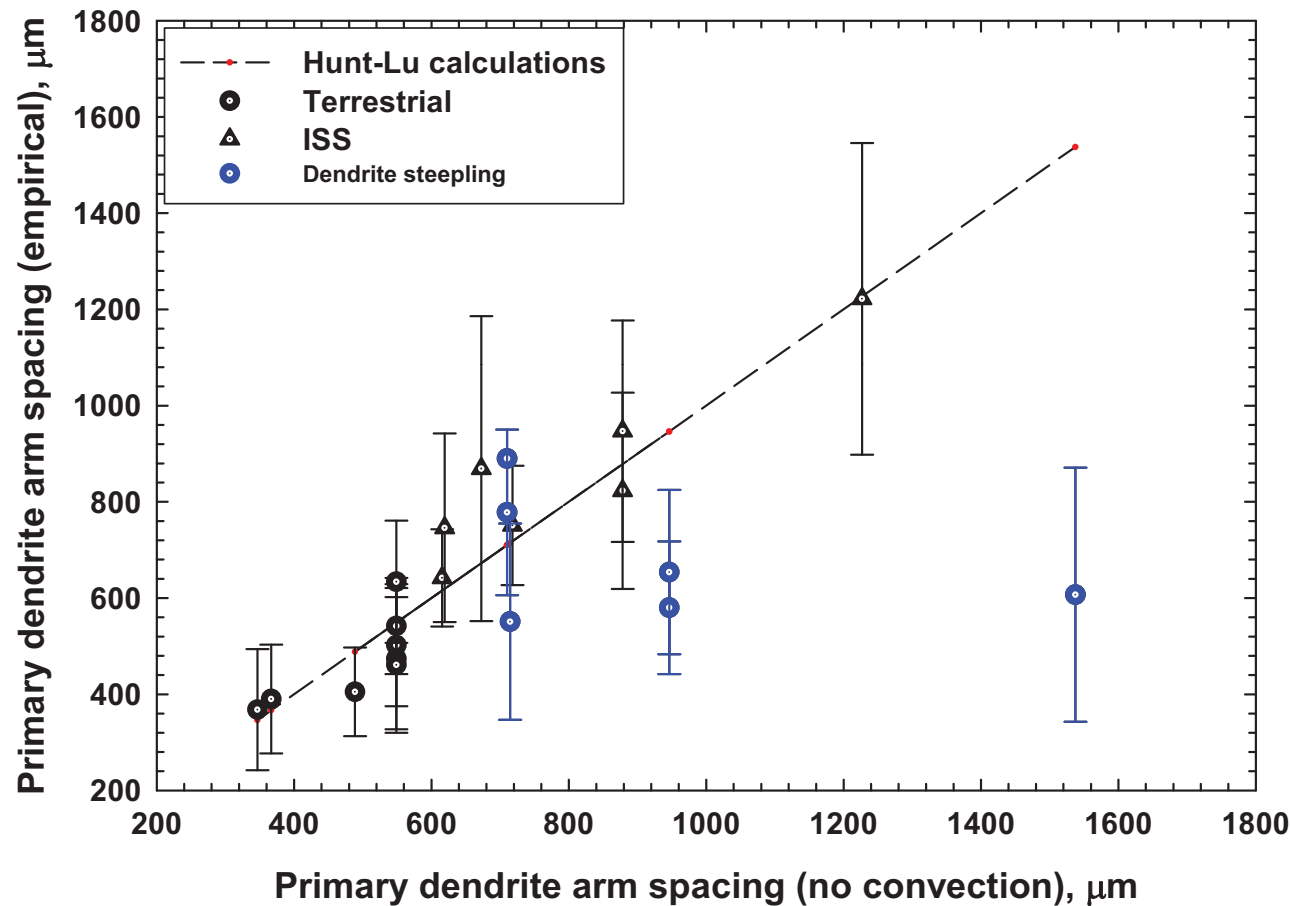
1. Secondary arm melts back because of its curvature.
2. Mass of the melted arm deposits on trunk surface where there is negative curvature.



$$\phi^3 = 96 \frac{D_l \Gamma}{R G (1 - k)} \ln \left\{ \frac{\left(1 + \frac{R G t}{m_l C_o} \right)}{\left(1 + \frac{R G t_o}{m_l C_o} \right)} \right\} + \phi_0^3$$

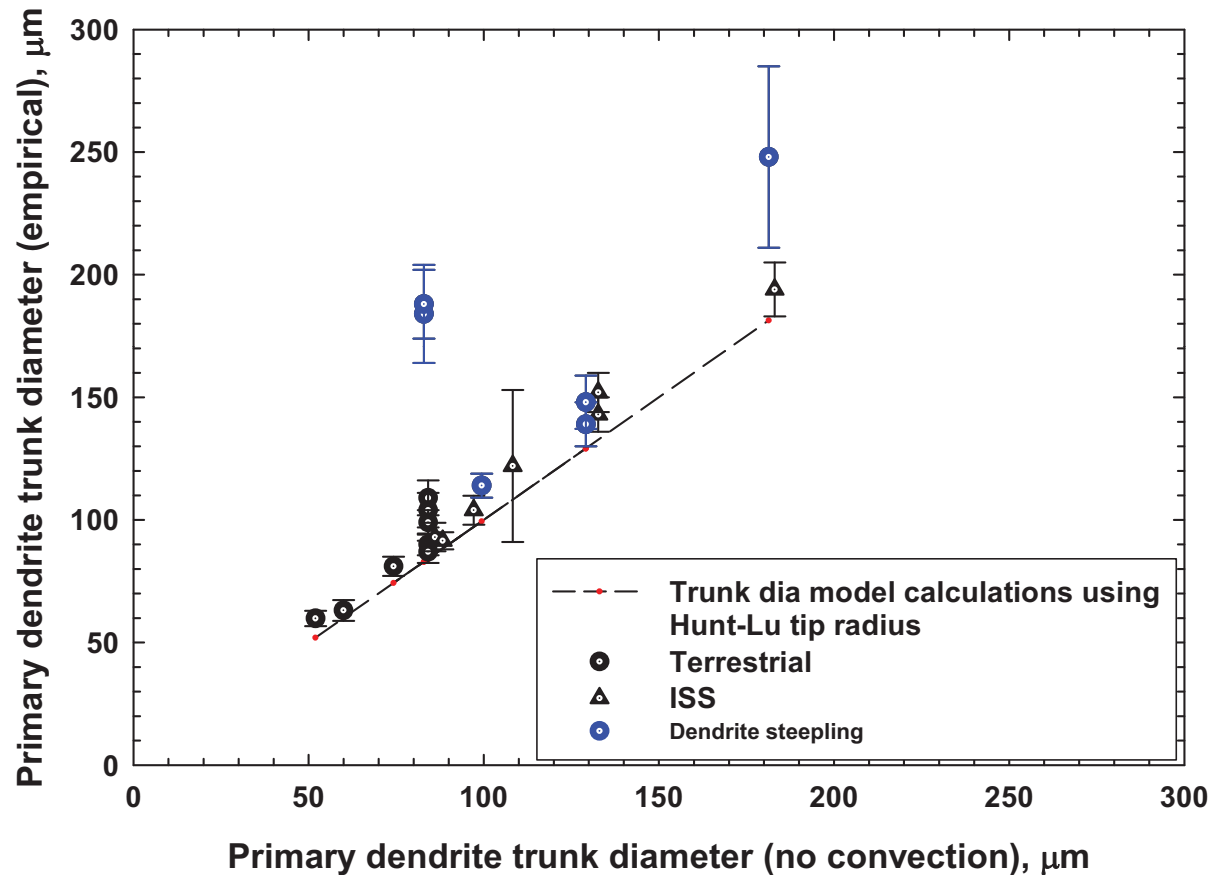
Mushy Zone Freezing Time $\sim m_l(C_E - C_o)/RG_m$

Primary dendrite arm spacing compared with Hunt-Lu Model



- ISS-DS: Good agreement with predictions from Hunt-Lu model.
- Terrestrial DS ("Not steeped") : Good agreement with predictions from Hunt-Lu model.
- Terrestrial DS ("steeped"): Convection decreases primary dendrite arm spacing.

Primary dendrite trunk diameter compared with trunk diameter model (using r_t (Hunt-Lu))



- ISS-DS: Good agreement with predictions from the trunk-diameter model.
- Terrestrial DS (“Not steeped”) : Good agreement with predictions from model.
- Terrestrial DS (“steeped”): Convection increases trunk diameter.

Trunk diameter from Dupouy et al. Micrographs (Al-26.5 Cu)

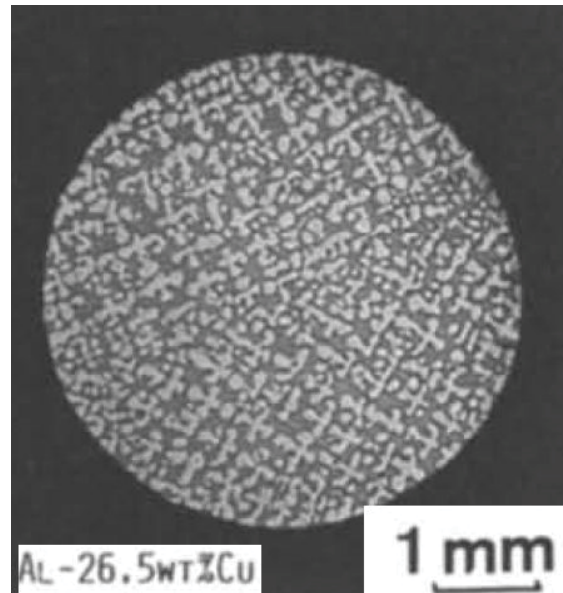
(M.D. Dupouy, D. Camel and J.J. Favier, Acta. Metall. Mater. Vol. 37, No. 4, pp. 1143-1157, 1989)

Al-26.5 wt% Cu, 30 K cm⁻¹,
4.2 μm s⁻¹



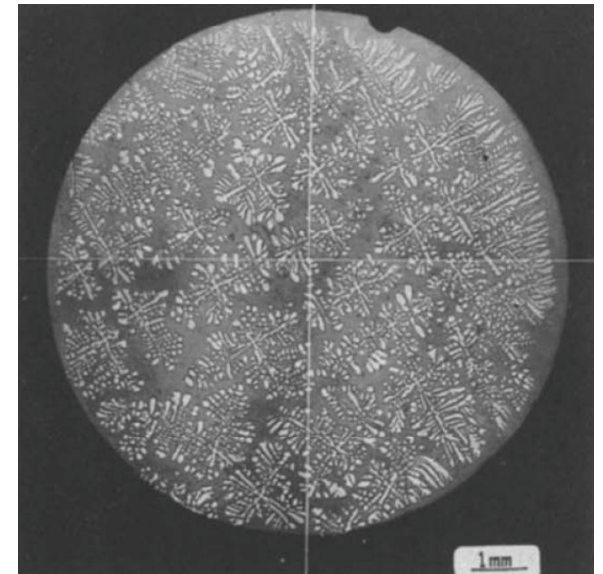
Terrestrial: Solutally stable,
thermally stable mode

Al-26.5 wt% Cu, 25 K cm⁻¹,
4.2 μm s⁻¹



Terrestrial: Solutally unstable,
thermally stable mode

Al-26.5 wt % Cu, 30 K cm⁻¹,
4.2 μm s⁻¹



Microgravity:

Trunk diameter → 120 ± 18 μm?

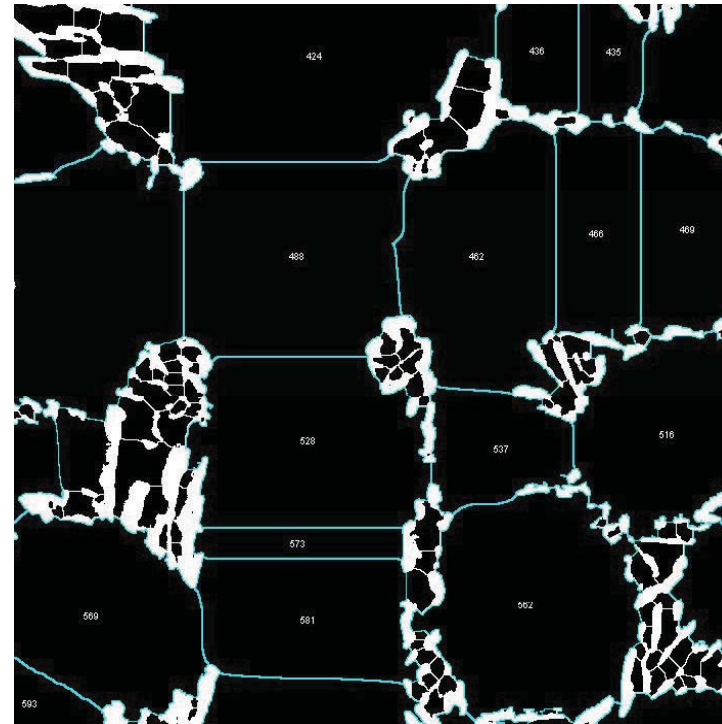
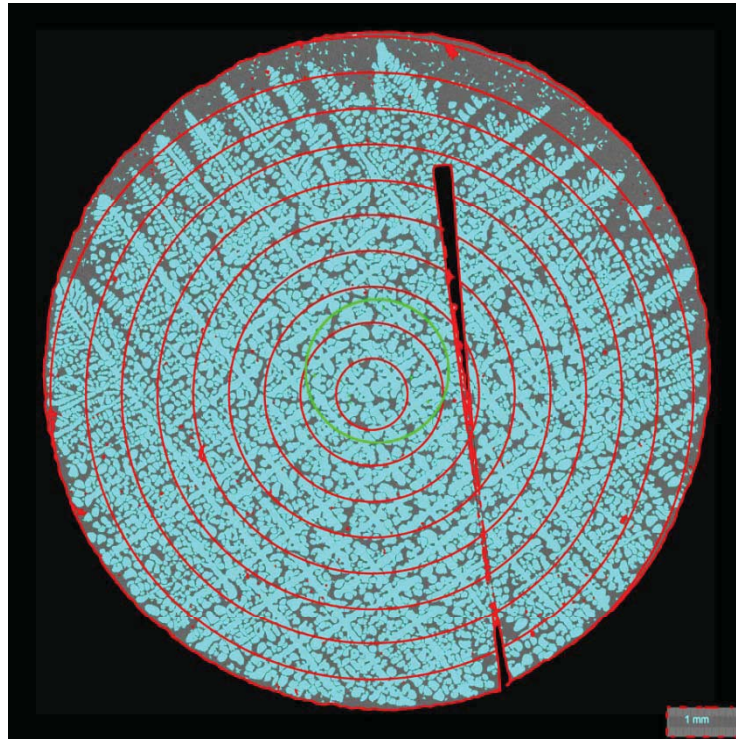
122 ± 18 μm?

92 ± 11 μm?

21

Natural convection appears to increase primary dendrite trunk diameter (Al-26.5 Cu)

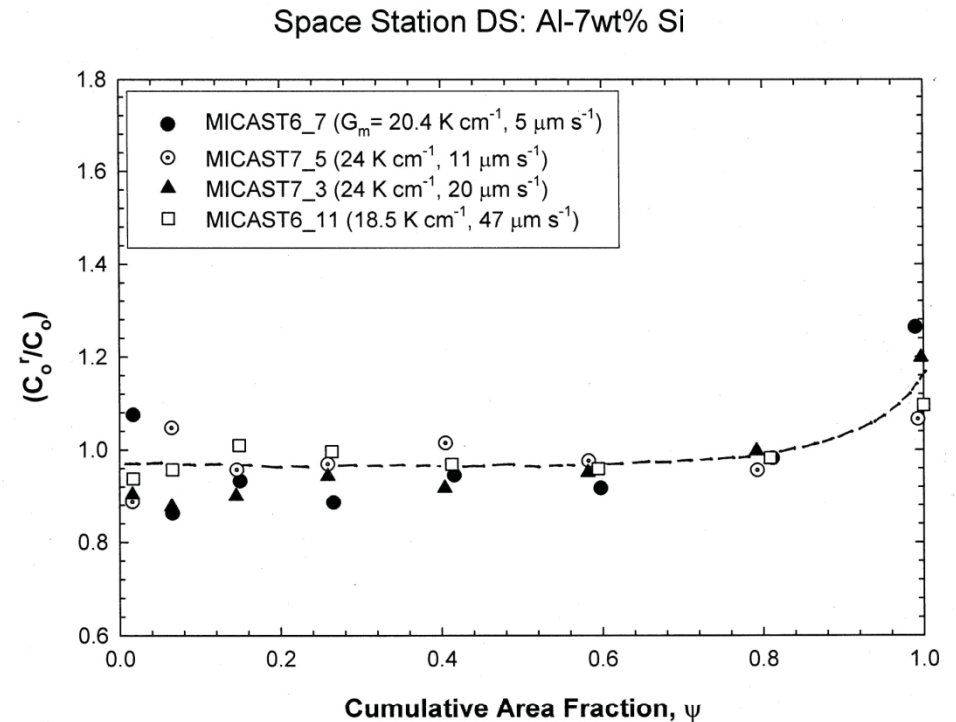
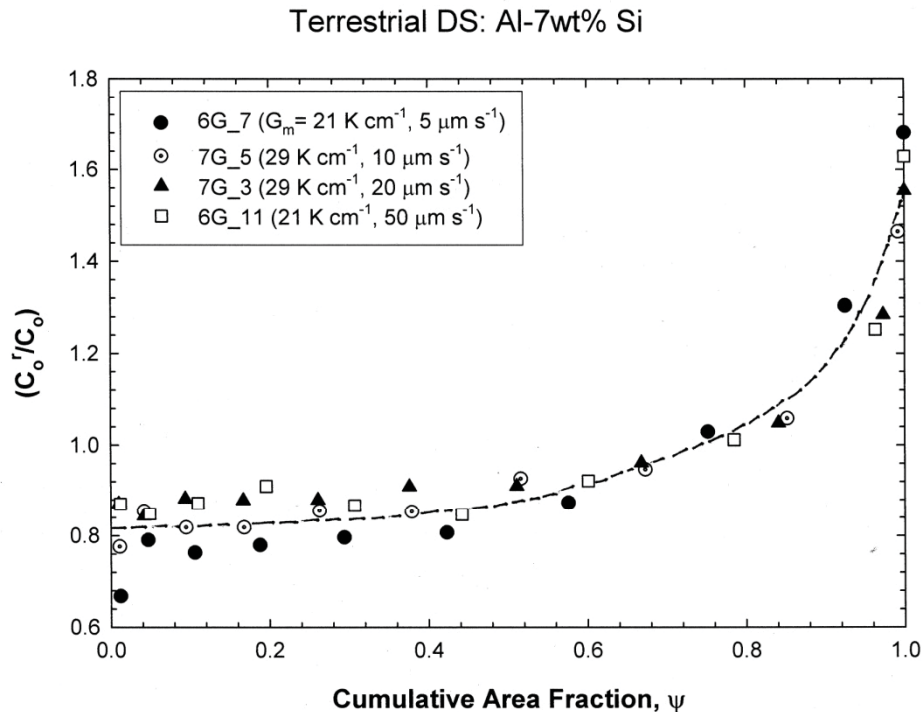
Radial macrosegregation measurement in DS Al-7% Si



Measure Fraction Eutectic (f_E) vs. Fraction Solidified (ψ) wrt steeple center of mass.

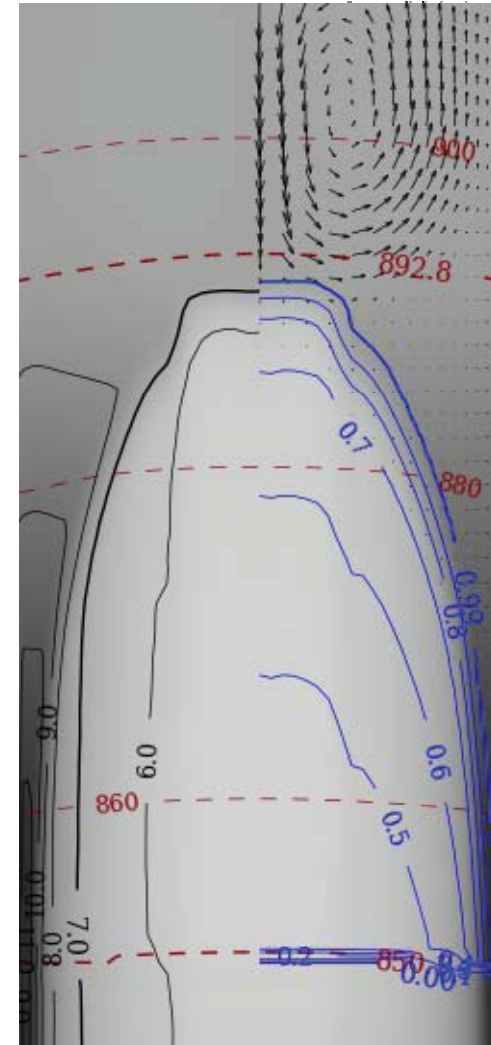
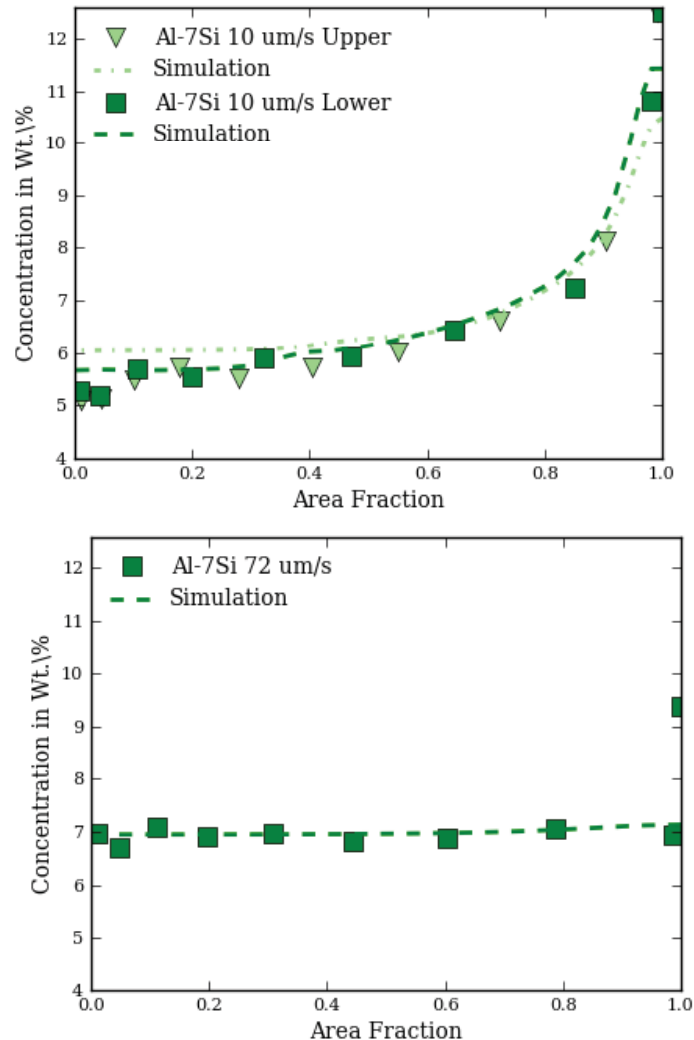
$$\text{Local Solute Content: } C_0^r = 1.094 + 11.60f_E$$

Comparison of radial macrosegregation: Al-7% Si directionally solidified on ground and on ISS



Extensive radial macrosegregation associated with “dendrite steeping” in terrestrial samples is nearly absent in ISS processed samples.

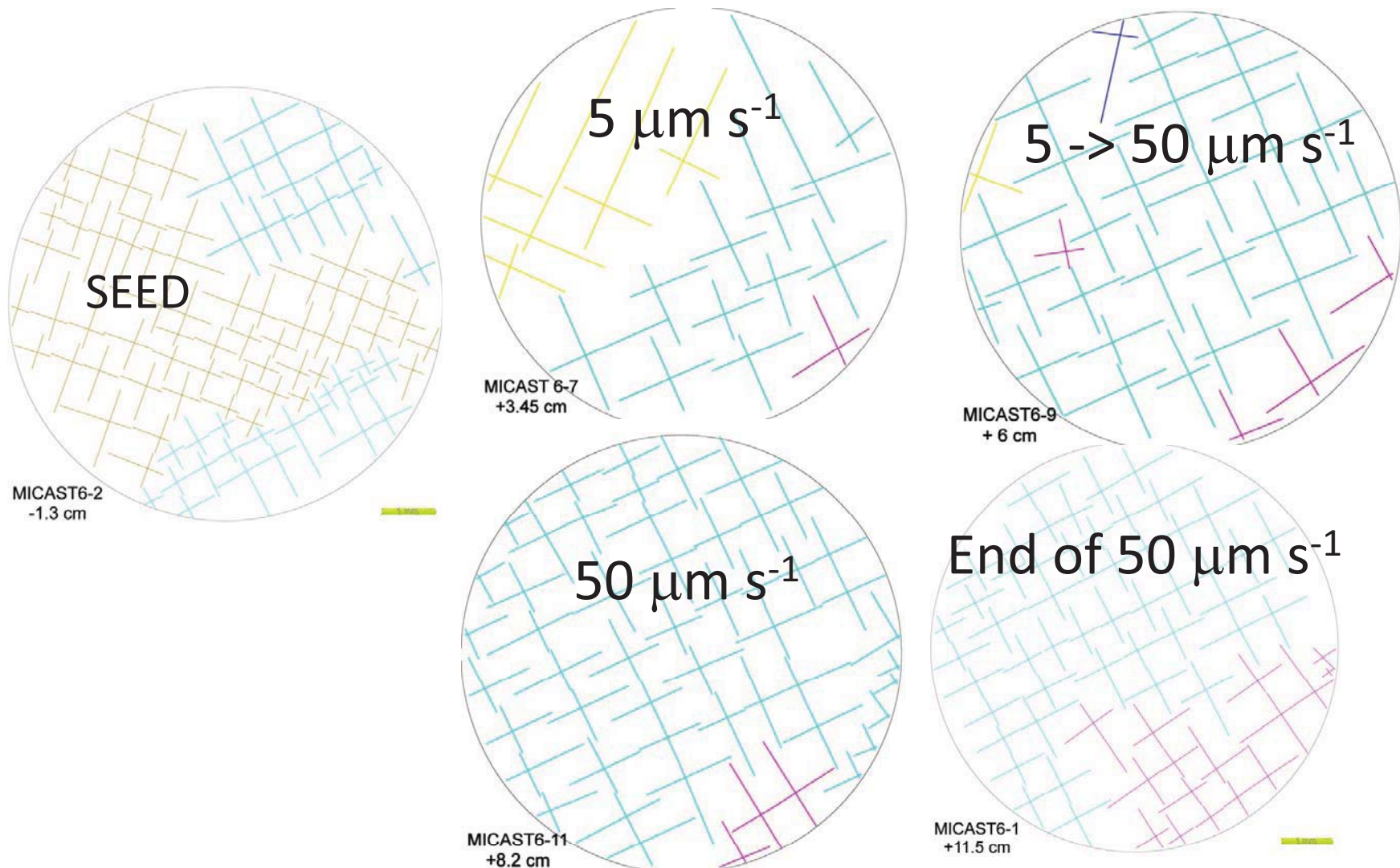
Numerical modeling of radial macrosegregation during directional solidification of Al-7Si alloy



Similar to Beckermann et al (88)
and Felicelli et al. (91)

Surprises??

MICAST-6: Grains elimination and formation along DS length



SPURIOUS GRAINS DURING DS IN THE ABSENCE OF CONVECTION??

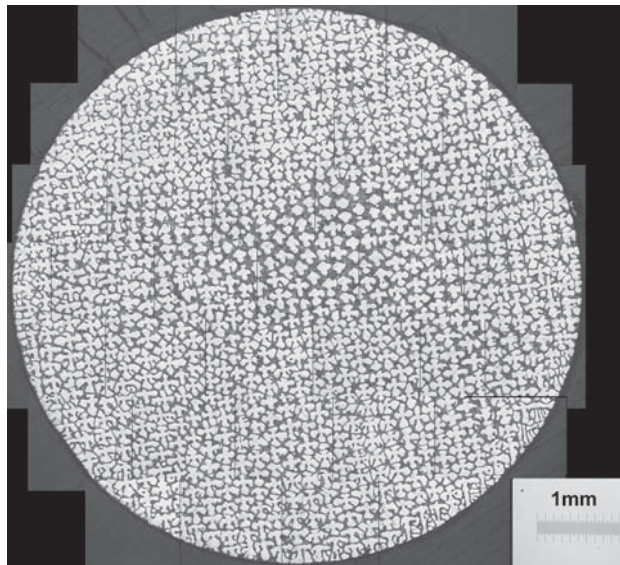
Conclusions

- Primary dendrite arm spacings of Al-7 wt% Si alloy directionally solidified in low gravity environment of space (MICAST-6 and MICAST-7: Thermal gradient ~ 19 to 26 K cm^{-1} , Growth speeds varying from 5 to $50 \mu\text{m s}^{-1}$) show a good agreement with predictions from Hunt-Lu and Trivedi models.
- Primary dendrite trunk diameters of the ISS processed samples show a good fit with a simple analytical model based on Kirkwood's approach, proposed here.
- Natural convection,
 - decreases primary dendrite arm spacing.
 - appears to increase primary dendrite trunk diameter.
 - produces radial macrosegregation.
- Spurious grains formed during DS in “low- $g < 10^{-4} \text{ g}$ ”??

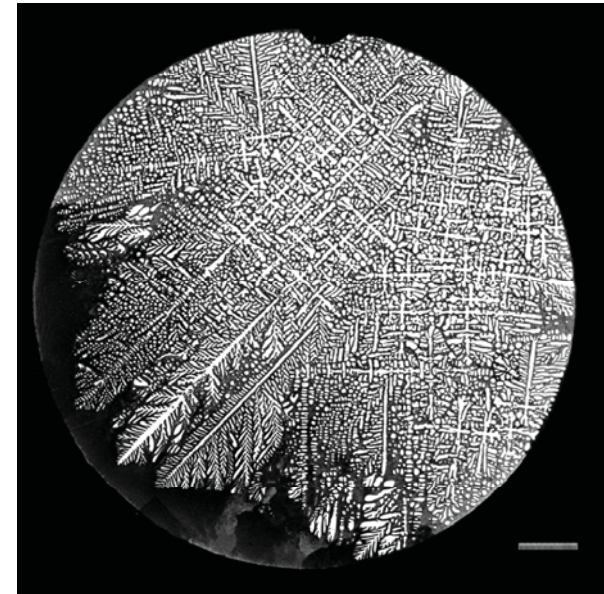
Can we predict microstructural inhomogeneity induced by natural convection during terrestrial DS of industrial components?

Processing parameters → Diffusive transport → Microstructure → Properties
• Well understood

(Pb-5.8Sb: 40 K cm⁻¹, 10 μms⁻¹)



(Al-19 Cu: 81 K cm⁻¹, 10 μms⁻¹)



Processing parameters → **Natural convection** → Microstructure
• Not understood

Grateful for support from

- NASA
- ESA
- DLR-MUSC
- ALCOA